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(71) Applicant (for all designated States except US): **DOW  
CORNING CORPORATION** [US/US]; 2200 West  
Salzburg Road, Midland, MI 48686-0994 (US).

(72) Inventors; and

(75) Inventors/Applicants (for US only): **GORDON, Glenn**,  
**Viaplana** [US/US]; 3714 Collingwood Street, Midland,  
MI 48642 (US). **SCHMIDT, Randall, Gene** [US/US];  
813 Honeysuckle Circle, Midland, MI 48642 (US).  
**STARK-KASLEY, Lori, Ann** [US/US]; 2683 East  
Mier Road, Midland, MI 48642 (US). **WIEBER, Gary**,  
**Michael** [US/US]; 4407 Huron Drive, Midland, MI 48642  
(US).

(74) Common Representative: **ZOMBECK, Alan**; IP De-  
partment - Mail CO1232, Dow Corning Corporation, 2200  
West Salzburg Road, Midland, MI 48686-0994 (US).

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(54) Title: MQ AND T-PROPYL SILOXANE RESINS COMPOSITIONS

(57) Abstract: Siloxane resin compositions obtained by mixing a MQ siloxane resin with a propyl silsesquioxane resin are disclosed. These siloxane resins are useful in a variety of personal, household, automotive and medical care applications, and in particular, as a resin additive in pigmented cosmetic formulations.

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## MQ AND T-PROPYL SILOXANE RESINS COMPOSITIONS

## CROSS REFERENCE TO RELATED APPLICATIONS

[0001] Not applicable.

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## FIELD OF THE INVENTION

[0002] The present invention provides siloxane resin compositions obtained by mixing a MQ siloxane resin with a propyl silsesquioxane resin. The siloxane resin compositions of the present invention are useful in a variety of personal, household, automotive and medical care applications, and in particular, as a resin additive in pigmented cosmetic formulations.

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## BACKGROUND OF THE INVENTION

[0003] Siloxane resins of the general formula  $R_nSiO_{(4-n)/2}$ , where R is an alkyl group and n is generally less than 1.8, are an important family of silicone polymers because of their utility in many commercial applications such as adhesive compositions and coatings applications. One particular subclass of siloxane resins, known as MQ resins (since they comprise primarily of “M” units of the general formula  $R_3SiO_{1/2}$  and “Q” units of the general formula  $SiO_2$ ), have found utility in cosmetic formulations. In particular MQ resins are commonly used in “extended wear” or “transfer resistant” cosmetic formulations. In these formulations, MQ resins enhance the substantivity of the pigments or other formulation actives to skin after application creating a longer lasting, and hence extended wear product.

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[0004] Representative examples of transfer resistant cosmetic compositions using MQ resins are found in US 6,071,503, US 6,074,654, US 6,139,823, US 6,340,466, WO 97/17058, and WO 97/17059 which disclose compositions comprising the combination of organosiloxane resins and fluid diorganosiloxane resins with a volatile carrier.

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[0005] US 5,330,747 teaches cosmetics with enhanced durability using a film forming agent from a pressure sensitive adhesive composition comprising a trimethylsilyl endblocked resinous copolymer, a silanol endblocked polydiorganosiloxane fluid, and a phenyl containing polysiloxane fluid.

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[0006] US 5,075,103 and US 5,733,537 teach a hair treating method for imparting curl retention to hair in which at least one film-forming ingredient is applied to the hair. The

improvement utilizes as the film-forming ingredient an organosilicon compound which is a nonpolar silsesquioxane.

[0007] US 5,800,816 discloses cosmetic compositions having improved transfer resistance comprising: a) from about 0.1-60% by weight of trimethylated silica, b) from about 0.1-60%  
5 by weight of a volatile solvent having a viscosity of 0.5 to 100 centipoise at 25°C, c) 0.1-60% by weight of a nonvolatile oil having a viscosity of 200 to 1,000,000 centipoise at 25°C, d) 0.1-80% of a cosmetically acceptable carrier.

[0008] US 5,837,223 and US 6,036,947 teach transfer resistant high luster cosmetic stick compositions comprising, by weight of the total composition: a) 10-70% of a volatile solvent  
10 having a viscosity of 0.5 to 20 centipoise at 25.degree. C., b) 0.5-40% of a guerbet ester, and c) 0.1-20% of a siloxysilicate polymer.

[0009] GB 2,319,527 discloses fragrance releasing non-volatile polysiloxanes based on a high molecular weight polydiorganosiloxane compounds where at least one or more of the organic substituents of the polymer is a radical derived from a fragrant alcohol.

15 [0010] Japanese examined patent publication 1994-72085 teaches makeup cosmetic compositions having improved water resistance and durability containing an organic silicone resin, a volatile silicone oil, and a make up powder.

[0011] While the use of MQ resins in cosmetics have led to formulations having extended wear or transfer resistance, a need exists to alter the properties of the siloxane resins used in  
20 such formulations. In particular, films of MQ resins used in these formulations can have a matte finish and are brittle. Thus, there is a need for improved siloxane resins that offer at least comparable extended wear and transfer resistance properties of the MQ resins presently used in cosmetic formulations, but having improved gloss (i.e. non-matte), are still non-tacky, and are more flexible. Furthermore, there is a need for resins in hair care formulations that  
25 improve the curl retention properties of hair following treatment.

[0012] The present inventors have discovered improved siloxane resin compositions by forming compatible blends of propyl silsesquioxane resins with MQ resins. The resulting siloxane resin compositions have improved physical properties. In particular, cosmetic formulations containing the present siloxane resin compositions have improved gloss and are

more flexible than MQ resins alone, while maintaining their long lasting or wear characteristics.

#### SUMMARY OF THE INVENTION

[0013] This invention provides a siloxane resin composition obtained by mixing:

- 5           A) a MQ resin comprising at least 80 mole % of siloxy units selected from  $(R^1_3SiO_{1/2})_a$  and  $(SiO_{4/2})_b$  units, where  $R^1$  is independently an alkyl group having from 1 to 8 carbon atoms, an aryl group, a carbinol group, or an amino group, with the proviso that at least 95 mole % of the  $R^1$  groups are alkyl groups, a and b has a value greater than zero, and the ratio of a/b is 0.5 to 1.5;
- 10           B) a propyl silsesquioxane resin comprising at least 80 mole %  $R^3SiO_{3/2}$  units, where  $R^3$  is independently an alkyl group having from 1 to 8 carbon atoms, an aryl group, a carbinol group, or an amino group, with the proviso that at least 40 mole % of the  $R^3$  groups are propyl, and optionally;
- 15           C) a volatile siloxane or organic solvent, wherein the weight ratio of component A to B is from 1:99 to 99:1.

[0014] The siloxane resins are useful in a variety of personal, household, or medical care compositions. In particular, the siloxane resin compositions provide glossy, non-tacky films that can be used to enhance the substantivity of color cosmetic formulations. The siloxane resin compositions can also be used in hair care formulations to enhance curl retention properties. The siloxane resin compositions can also enhance the feel of treated hair, providing a softer feel compared to other siloxane resin. Thus, the present invention provides personal, household, automotive, or medical care compositions comprising the siloxane resin compositions described herein.

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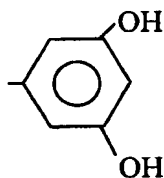
#### DETAILED DESCRIPTION OF THE INVENTION

[0015] Component A) is a MQ resin comprising at least 80 mole% of siloxy units selected from  $(R^1_3SiO_{1/2})_a$  and  $(SiO_{4/2})_b$  units, where  $R^1$  is an alkyl group having from 1 to 8 carbon atoms, an aryl group, a carbinol group, or an amino group, with the proviso that at least 95 mole % of the  $R^1$  groups are alkyl groups, a and b has a value greater than zero, and the ratio of a/b is 0.5 to 1.5.

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[0016] The  $R^1$  units of the MQ resin are independently an alkyl group having from 1 to 8 carbon atoms, an aryl group, a carbinol group, or an amino group. The alkyl groups are illustrated by methyl, ethyl, propyl, butyl, pentyl, hexyl, and octyl. The aryl groups are illustrated by phenyl, naphthyl, benzyl, tolyl, xylyl, xenyl, methylphenyl, 2-phenylethyl, 2-phenyl-2-methylethyl, chlorophenyl, bromophenyl and fluorophenyl with the aryl group typically being phenyl.

[0017] For the purposes of this invention a "carbinol group" is defined as any group containing at least one carbon-bonded hydroxyl (COH) radical. Thus the carbinol groups may contain more than one COH radical such as for example



[0018] The carbinol group if free of aryl groups has at least 3 carbon atoms, or an aryl-containing carbinol group having at least 6 carbon atoms. The carbinol group free of aryl groups having at least 3 carbon atoms is illustrated by groups having the formula  $R^4OH$  wherein  $R^4$  is a divalent hydrocarbon radical having at least 3 carbon atoms or divalent hydrocarbonoxy radical having at least 3 carbon atoms. The group  $R^4$  is illustrated by alkylene radicals such as  $-(CH_2)_x-$  where x has a value of 3 to 10,

$-CH_2CH(CH_3)-$ ,  $-CH_2CH(CH_3)CH_2-$ ,  $-CH_2CH_2CH(CH_2CH_3)CH_2CH_2CH_2-$ , and  $-OCH(CH_3)(CH_2)_x-$  wherein x has a value of 1 to 10.

[0019] The aryl-containing carbinol group having at least 6 carbon atoms is illustrated

by groups having the formula  $R^5OH$  wherein  $R^5$  is an arylene radical such as  $-(CH_2)_xC_6H_4-$  wherein x has a value of 0 to 10,  $-CH_2CH(CH_3)(CH_2)_xC_6H_4-$  wherein x has a value of 0 to 10,  $-(CH_2)_xC_6H_4(CH_2)_x-$  wherein x has a value of 1 to 10. The aryl-containing carbinol groups typically have from 6 to 14 atoms.

[0020] The amino group is illustrated by groups having the formula  $-R^6NH_2$  or  $-$

$R^6NHR^7NH_2$  wherein  $R^6$  is a divalent hydrocarbon radical having at least 2 carbon atoms and  $R^7$  is a divalent hydrocarbon radical having at least 2 carbon atoms. The group  $R^6$  is

typically an alkylene radical having from 2 to 20 carbon atoms.  $R^6$  is illustrated by ethylene, propylene,  $-\text{CH}_2\text{CHCH}_3-$ , butylene,  $-\text{CH}_2\text{CH}(\text{CH}_3)\text{CH}_2-$ , pentamethylene, hexamethylene, 3-ethyl-hexamethylene, octamethylene, and decamethylene.

[0021]  $R^7$  is typically an alkylene radical having from 2 to 20 carbon atoms.  $R^7$  is

illustrated by ethylene, propylene,  $-\text{CH}_2\text{CHCH}_3-$ , butylene,  $-\text{CH}_2\text{CH}(\text{CH}_3)\text{CH}_2-$ , pentamethylene, hexamethylene, 3-ethyl-hexamethylene, octamethylene, and decamethylene.

[0022] Typical amino groups are  $-\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2$  and

$-\text{CH}_2(\text{CH}_3)\text{CHCH}_2(\text{H})\text{NCH}_3$ ,  $-\text{CH}_2\text{CH}_2\text{NHCH}_2\text{CH}_2\text{NH}_2$ ,  $-\text{CH}_2\text{CH}_2\text{NH}_2$ ,

$-\text{CH}_2\text{CH}_2\text{NHCH}_3$ ,  $-\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{NH}_2$ ,  $-(\text{CH}_2\text{CH}_2\text{NH})_3\text{H}$ , and

$-\text{CH}_2\text{CH}_2\text{NHCH}_2\text{CH}_2\text{NHC}_4\text{H}_9$ .

[0023] Typically,  $R^1$  is a methyl group.

[0024] MQ resins suitable for use as component (A), and methods for their preparation, are known in the art. For example, U.S. Patent No. 2,814,601 to Currie et al., November 26, 1957, which is hereby incorporated by reference, discloses that MQ resins can be prepared by converting a water-soluble silicate into a silicic acid monomer or silicic acid oligomer using an acid. When adequate polymerization has been achieved, the resin is end-capped with trimethylchlorosilane to yield the MQ resin. Another method for preparing MQ resins is disclosed in U.S. Patent No. 2,857,356 to Goodwin, October 21, 1958, which is hereby incorporated by reference. Goodwin discloses a method for the preparation of an MQ resin by the cohydrolysis of a mixture of an alkyl silicate and a hydrolyzable trialkylsilane organopolysiloxane with water.

[0025] The MQ resins suitable as component (A) in the present invention may contain D and T units, providing that at least 80 mole %, alternatively 90 mole % of the total siloxane units are M and Q units. The MQ resins may also contain hydroxy groups. Typically, the MQ resins have a total weight % hydroxy content of 2-10 weight %, alternatively 2-5 weight %. The MQ resins can also be further "capped" wherein residual hydroxy groups are reacted with additional M groups.

[0026] Component (B) is a propyl silsesquioxane resin comprising at least 80 mole % of  $\text{R}^3\text{SiO}_{3/2}$  units, where  $\text{R}^3$  is independently an alkyl group having from 1 to 8 carbon atoms an aryl group, a carbinol group, or an amino group, and with the proviso that at least 40 mole % of the  $\text{R}^3$  groups are propyl. The propyl silsesquioxane resins are referred herein as T-propyl

resins, since the majority of the siloxane units are T units of the general formula  $R^3SiO_{3/2}$  where at least 40 mole %, alternatively 50 mole %, or alternatively 90 mole % of the  $R^3$  groups are propyl. The  $R^3$  groups are illustrated by the same structures as described above for  $R^1$  groups.

5 [0027] Silsesquioxane resins are well known in the art and are typically prepared by hydrolyzing an organosilane having three hydrolyzable groups on the silicon atom, such as a halogen or alkoxy group. Thus, component (B) can be obtained by hydrolyzing propyltrimethoxysilane, propyltriethoxysilane, propyltripropoxysilane, or by co-hydrolyzing the aforementioned propylalkoxysilanes with various alkoxysilanes. Examples of these  
10 alkoxysilanes include methyltrimethoxysilane, methyltriethoxysilane, methyltriisopropoxysilane, dimethyldimethoxysilane, and phenyltrimethoxysilane. Propyltrichlorosilane can also be hydrolyzed alone, or in the presence of alcohol. In this case, co-hydrolyzation can be carried out by adding methyltrichlorosilane, dimethyldichlorosilane, phenyltrichlorosilane, or similar chlorosilanes and methyltrimethoxysilane,  
15 methyltriethoxysilane, methyltriisopropoxysilane, or similar methylalkoxysilane. Alcohols suitable for these purposes include methanol, ethanol, n-propyl alcohol, isopropyl alcohol, butanol, methoxy ethanol, ethoxy ethanol, or similar alcohols. Examples of hydrocarbon-type solvents which can also be concurrently used include toluene, xylene, or similar aromatic hydrocarbons; hexane, heptane, isooctane, or similar linear or partially branched saturated  
20 hydrocarbons; and cyclohexane, or similar aliphatic hydrocarbons.

[0028] The T-propyl resins suitable as component B) in the present invention may contain M, D, and Q units, providing that at least 80 mole %, alternatively 90 mole % of the total siloxane units are T units. The T-propyl resins may also contain hydroxy and/or alkoxy groups. Typically, the T-propyl resins have a total weight % hydroxy content of 2-10 weight % and a  
25 total weight % alkoxy content of up to 20 weight %, alternatively 6-8 weight% hydroxy content and up to 10 weight % alkoxy content.

[0029] A volatile siloxane or organic solvent can be included as optional component C) when mixing components A) and B). Any volatile siloxane or organic solvent can be selected providing components A) and B) are miscible with the solvent.

30 [0030] The volatile siloxane solvent can be a cyclic polysiloxane, a linear polysiloxane, or mixtures thereof. Some representative volatile linear polysiloxanes are hexamethyldisiloxane, octamethyltrisiloxane, decamethyltetrasiloxane, tetradecamethylhexasiloxane, and

hexadecamethylheptasiloxane. Some representative volatile cyclic polysiloxanes are hexamethylcyclotrisiloxane, octamethylcyclotetrasiloxane, decamethylcyclopentasiloxane, and dodecamethylcyclohexasiloxane. The organic solvent can be an alcohol such as methanol, ethanol, isopropanol, butanol, or n-propanol, a ketone such as acetone, methylethyl ketone, or methyl isobutyl ketone; an aromatic hydrocarbon such as benzene, toluene, or xylene; an aliphatic hydrocarbon such as heptane, hexane, octane, or isododecane; a glycol ether such as propylene glycol methyl ether, dipropylene glycol methyl ether, propylene glycol n-butyl ether, propylene glycol n-propyl ether, or ethylene glycol n-butyl ether, a halogenated hydrocarbon such as dichloromethane, 1,1,1-trichloroethane or methylene chloride, chloroform, dimethyl sulfoxide, dimethyl formamide, acetonitrile, tetrahydrofuran, white spirits, mineral spirits, or naphtha, or an ester.

[0031] There are no special requirements or conditions needed for effecting the mixing of components A) and B). Thus, any method in the art known to effect mixing of such compositions can be used. Components A) and B) can be optionally contained in a solvent, as described supra as component C). The mixing can be conducted in a batch, semi-continuous, or continuous process.

[0032] The weight ratio of component A) to component B) (i.e. A/B) in the mixture can vary from 99:1 to 1:99, alternatively 85:15 to 15:85.

[0033] The siloxane resin compositions of the present invention are useful in a variety of personal, household, automotive, or medical care applications. In particular, the siloxane resin compositions of the present invention provide glossy, non-tacky films that can be used to enhance the substantivity of color cosmetic formulations. The siloxane resin compositions can also be used as additives in hair care formulations to enhance curl retention properties. Thus, the present invention provides personal, household, automotive, or medical care compositions comprising the siloxane resins described herein.

## EXAMPLES

[0034] The following examples are presented to further illustrate the compositions and methods of this invention, but are not to be construed as limiting the invention. All parts and percentages in the examples are on a weight basis and all measurements were obtained at about 23°C, unless indicated to the contrary.



**Materials**

MQ Resin = a MQ resin having the formula  $M_{0.43}Q_{0.57}$  and  $M_n = 3230$  dissolved in xylenes at 70.8% solids. The MQ resin was prepared according to techniques taught by Daudt in

5 US 2,676,182.

T propyl resin = propyl silsesquioxane resin at 74.8 wt % in toluene. The propyl silsesquioxane resin was prepared from the hydrolysis of propyl trichlorosilane.

10 Phenyl silsesquioxane resin = a phenyl silsesquioxane solid flake resin at 100 wt % solids prepared from the hydrolysis of phenyl trichlorosilane.

**Examples 1-6**

- 15 [0035] Solutions of MQ resin and T propyl resin were mixed in a glass bottle by shaking or putting on a mixing wheel. An aliquot of each mixture was poured into a 2-inch diameter aluminum weighing dish and heated in a forced air oven for one hour at 110°C followed by one hour and 25 minutes at 140°C. Qualitative visual observations of clarity, brittleness and hardness were made (Table 1).
- 20 [0036] These examples demonstrate the unexpected miscibility of the MQ and T propyl resins, based on devolatilized blend clarity and the increasing hardness and brittleness trend as the MQ resin loading increased.

Table 1

MQ/T propyl resin blends.

Example	MQ (g)	T propyl (g)	Aliquot (g)	Dried Sample (g)	MQ loading (wt%)	Clarity	Qualitative Brittleness	Qualitative Hardness
1	0.00	13.46	2.0475	1.5361	0.0%	clear	Does not crack	Gummy soft solid
2	1.40	12.03	2.0643	1.5415	9.9%	clear	Slight cracking on extreme	"Grabby" soft solid
3	2.88	16.74	2.0840	1.5517	14.0%	clear	Cracks on moderate	Harder than #2
4	4.19	9.39	2.0746	1.5414	29.7%	clear	Cracks on moderate	Harder than #3
5	5.72	8.14	2.1066	1.5606	39.9%	clear	Cracks on slight	Harder than #4
6	7.11	6.82	2.0257	1.4968	49.6%	clear	Cracked upon	similar to #5

[0001] Rheology of MQ/T(Pr) blends showed a single softening point and steady change in softening temperature based on blend composition, as show in figures 1 and 2 below.

**Comparative Examples 7-19**

5 ***MQ/DT(Me)/T(Pr) Resin Blend Comparisons***

[0002] Solutions of MQ resins having various M:Q ratios of 0.75, 0.85, 0.95 (59.9, 73.3 and 70.8 wt% in xylenes respectively), a siloxane resin having the formula  $D_{0.15}^{Me2} T_{0.85}^{Me}$  (50.3 wt% in toluene) was first prepared and then mixed with T propyl resin (75.5 wt% in toluene) were mixed. An aliquot of each mixture was poured into a 2-inch diameter aluminum weighing dish and heated in a forced air oven overnight at 105°C followed by one hour and 30 minutes at 150°C. Qualitative visual observations of clarity, brittleness and hardness made are summarized in Table 2.

10

[0039] These examples demonstrate the immiscibility of MQ resins with the DT methyl resin and with DT methyl resin/propylsilsesquioxane hydrolyzate resin blends.

5

Table 2

## MQ/Methyl-T resin blends

Example	M:Q Ratio of MQ Resin	MQ/DT(Me)/T(Pr) Blend Composition**	Clarity	Qualitative Brittleness
7	0.75	10/90/0	Hazy	Brittle
8	0.75	25/75/0	Hazy	Brittle
9	0.75	50/50/0	Hazy	Brittle
10	0.85	10/90/0	Hazy	Brittle
11	0.85	25/75/0	Hazy	Brittle
12	0.85	50/50/0	Hazy	Brittle
13	0.95	10/90/0	Hazy	Brittle
14	0.95	25/75/0	Hazy	Brittle
15	0.95	50/50/0	Hazy	Brittle
16		0/50/50	Hazy	Flexible
17		0/75/25	Hazy	Leathery
18	0.85	25/25/50	Hazy*	Brittle
19	0.85	33/33/33	Hazy*	Brittle

\* sample clear at 150°C but becomes hazy on cooling to room temperature

\*\* DT(Me) = D<sub>0.15</sub><sup>Me2</sup> T<sub>0.85</sub><sup>Me</sup> resin and T(Pr) = T<sub>1.0</sub><sup>Pr</sup> hydrolyzate

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**Example 20 - 25***MQ:T<sup>Pr</sup> Blends in personal care formulations*

[0040] MQ resin, T-Propyl resin, and decamethylcyclopentasiloxane were mixed in a glass bottle by shaking and placing on a mixing wheel at the ratios indicated in Table 3 until homogeneous and uniform.

**Table 3****Formulation of MQ:TPr blends**

Example #	Wt % MQ solids	Wt% T <sup>Pr</sup> solids
20	100	0
21	0	100
22	85	15
23	50	50
24	15	85
25	30	70

[0041] Films resulting from the siloxane resins of Examples 20 – 25 were evaluated for gloss and tack at 35% solids in volatile solvent using a Leneta chart, evaluated in a foundation (color cosmetic) for durability, and as a hair fixative for curl retention and conditioning. The results are summarized in Table 4. The siloxane resins were applied to the hair tresses as 6 wt. % solutions in decamethylcyclopentasiloxane or isododecane carriers. Descriptions of the specific test methods and composition of the cosmetic formulation used in these evaluations follows Table 4.

Table 4

Example #	60° Gloss	Tack	Foundation Durability - $\Delta E$ (Change in color)	Curl Retention - Hair - % curl maintained after 5 hours at 95% RH
No Resin - neg control (exposed to carrier only) comparison	55	NA	9.2	30.5 -36.8, average to difficult to comb
20 (comparative)	34.8	Not tacky	3.8	53-58, Stiff rough, difficult to comb
21 (comparative)	78.4	Very tacky	11.0	44, Soft, sticky, easy to comb
22	37.5	Not tacky	2.8	48.7, Slightly rough, slightly sticky, average combability
23	44.6	Yes, Less with time	6.6	43.7, Slightly rough, slightly sticky, average combability
24	62.8	tacky		43.4, Sticky, shiny, average combability
25			8.3	

**Gloss Measurement:**

- 1) Coat Leneta charts (Form N2C) with solution using a #8 Meyer rod
- 2) Allow chart to dry for 1 hour. Measure 60° gloss using a portable gloss meter at 3 points on the left 1/3 of the chart. Calculate the average of the 3 gloss values. Evaluate coating for tack, greasiness, fingerprint mark and if and how the coating rubs off the chart.
- 3) 4 hours after the drawdown was completed, measure 60° gloss using a portable gloss meter at 3 points on the middle 1/3 of the chart. Calculate the average of the 3 gloss values. Evaluate coating for tack, greasiness, fingerprint mark and if and how the coating rubs off the chart.
- 4) 24 hours after the drawdown was completed, measure 60° gloss using a portable gloss meter at 3 points on the right 1/3 of the chart. Calculate the average of the 3 gloss values. Evaluate coating for tack, greasiness, fingerprint mark and if and how the coating rubs off the chart.
- 5) Using the average gloss at the 3 different times, calculate the overall average.

**Curl Retention Test Method****Materials**

- Prepared natural virgin brown hair tresses or oriental hair of 2 g, 25 cm.
- Comb Trade Mark : Ace,.
- Humidity chamber to regulate temperature and humidity during test.

**Procedure for pre-treating the swatches (washing):**

- 1) Wet 5 tresses for 30 sec with tap water at 37°C.
- 2) Lather the 5 tresses for 30 sec. With 5 g of the 30% SLS solution (Empicol LX28/Albright & Wilson), stroking the tresses downward, ensure you repeat the same movement for all the hair washed. Leave on hair for 30 sec.
- 3) Rinse the tresses for 1 minute with tap water at 37°C.
- 4) Remove the excess of water by running the tresses between the two fingers 3 times.
- 5) Allow the tresses to dry overnight on a paper towel at room temperature.

**Procedure for treating the swatches with the resin and curling:**

Blank or negative control is the solvent used in the treatments.

- 1) Dip 1 tress at a time 3 times in 37 °C tap water and remove excess of water by stroking the tress between 2 fingers
- 2) Lay the tress down on a clean support and apply 100 microliters of a 6% resins solution all along the using a calibrated micropipette
- 3) Detangle the tress completely
- 4) Roll the tress on rod spiral curler,
- 5) Leave the swatches to dry overnight in an oven at 40 °C.

**Test - curl retention measurements:**

1. Start the humidity chamber 2 hours before the test set at 70% humidity and 25 C.
2. Carefully remove the roller from hair by twisting it slightly, 10 minutes before the start of the test. Cut the ends of the tress in order to make it even (cut as few as possible).
- 5 3. Ensure each tress is correctly curled .
4. Hang the tresses in the humidity chamber : the bottom of the wax sealing on the tress should be on the line "0" of the millimeter paper sheet in the back of the chamber.
5. Measure hair tress length at predetermined intervals of time, the length is measured as the distance between the bottom of the wax sealing and the bottom of the tress - be aware that the bottom of the tress is going down, so the view angle is perpendicular to the glass..
- 10 6. After the 5 hours, remove the tresses from the humidity chamber and measure the tress length at its maximum, by unrolling it completely. Calculate curl retention as described below.

15 Percent curl retention is calculated as follows:

$$\% \text{ Curl Retention} = \frac{\text{max length} - \text{length at } T = x}{\text{max length} - \text{length at } T = 0} \times 100$$

20



**Foundation Formulation****Pigment Premix:**

50 wt% DC 245 Fluid

- 5 13.16 wt% Carde AS Titanium dioxide (caprylyl silane treated)
- 11.41 wt% Carde AS Red Iron Oxide (caprylyl silane treated)
- 18.26 wt% Carde AS Yellow Iron Oxide (caprylyl silane treated)
- 7.17 wt% Carde AS Black Iron Oxide (caprylyl silane treated)

10 **Procedure:**

- 1) Place DC 245 fluid in Waring Blender
- 2) Add titanium dioxide and mix by pressing the pulse button for 2 seconds for 15 seconds total.
- 3) Add red iron oxide and mix the same as titanium dioxide
- 15 4) Continue with the other pigments
- 5) When all materials have been dispersed, mix on high and shred for 30 sec to grind the pigments
- 6) Place premix into a round glass jar and place on pail roller for 6 hours.

20 **Phase A**

20.50 wt% Pigment Premix

7.50 wt% DC 5225C

8 wt% of a 50% resin solids in solvent

25 **Phase B**

54.80 wt % DI Water

1.0 wt% NaCl

0.20 wt% Polysorbate 20

30 **Procedure for Liquid Water in oil Foundation**

- 1) Put pigment dispersion on roller for 1 hour.
- 2) Weigh out resin and solvent to make a 50% solids dilution. Use oven and wheel to mix
- 3) Combine ingredients in Phase A, mix until uniform using a dual blade, turbulent style mixing action.
- 35 4) Combine ingredients in Phase B in separate beaker, mix until uniform using a magnetic stirrer
- 5) Increase mixing speed of Phase A to 1376 rpm and very slowly add Phase B through an addition funnel. This addition should take 10 mins.
- Continue mixing for an additional 10 min.

40

**Foundation Durability Method: Gardner Abrasion Tester**

- 1) Cut collagen into 3.5" x 3" pieces, place one on each of the 3" x 2.5" polycarbonate blocks and put in the humidity chamber overnight. This chamber must be at a constant 98% relative humidity level.
- 5 2) Remove collagen and block from chamber. Secure collagen to block with Scotch tape taking care not to place any tape on the top of the block's surface.
- 3) Add approximately 1 gram of foundation to the collagen, beading it across the top of the block. Using a #8 Meyer rod, coat the collagen with the foundation by placing the rod on the bead of foundation and spreading it downward to the bottom of the block. The final coating weight should be approximately 0.2 grams. This operation may need to be repeated to obtain the proper coating weight. Remove any material from the sides of the block.
- 10 4) Allow sample on collagen to dry. Drying times vary with different samples. Entire sample must be free from any wetness before testing. Measure color of sample on collagen for the initial baseline color using a spectrophotometer or colorimeter. L\*, a\*, and b\* designate the place of the colored object in a tri-dimensional space.
- 15 5) Place block with collagen face-up on the Gardner Abrasion Tester making sure that the block is in the tester. The soft side of Velcro is attached to the insult block to abrade or insult the foundation sample on the collagen. The insult block rubs back and forth across the foundation sample. One insult consists of one back and forth motion. Insult the foundation sample on the collagen 20 times. The machine can be stopped at certain intervals to measure the color.
- 20 6) After the foundation sample is insulted 20 times, the color is read as L\*, a\*, b\* and the change in color,  $\Delta E$ , is calculated (see equation below). The number of insults, coating weight, and repetitions can be changed to fit the needs of the material being tested. This is up to the discretion of the operator.
- 25

$\Delta L^*$ ,  $\Delta a^*$  and  $\Delta b^*$  = value after abrasion – value at initial baseline before abrasion.

$$\Delta E = (\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2})^{1/2}$$

30 With larger  $\Delta E$ 's, more foundation was removed and therefore the foundation is less durable.

**CLAIMS**

1. A siloxane resin composition obtained by mixing:

A) a MQ resin comprising at least 80 mole % of siloxy units selected from

( $R^1_3SiO_{1/2}$ )<sub>a</sub> and ( $SiO_{4/2}$ )<sub>b</sub> units, where  $R^1$  is independently an alkyl group having from 1 to 8 carbon atoms, an aryl group, a carbinol group, or an amino group, with the proviso that at least 95 mole % of the  $R^1$  groups are alkyl groups, a and b has a value greater than zero, and the ratio of a/b is 0.5 to 1.5;

B) a propyl silsesquioxane resin comprising at least 80 mole %  $R^3SiO_{3/2}$  units,

where  $R^3$  is independently an alkyl group having from 1 to 8 carbon atoms, an aryl group, a carbinol group, or an amino group,

with the proviso that at least 40 mole % of the  $R^3$  groups are propyl,

and optionally;

C) a volatile siloxane or organic solvent,

wherein the weight ratio of component A to B is from 1:99 to 99:1.

2. A personal care product comprising the siloxane resin of claim 1.

3. The personal care product of claim 2, where the personal care product is a cosmetic product.

4. The personal care product of claim 2, where the personal care product is a hair care product.

Figure 1

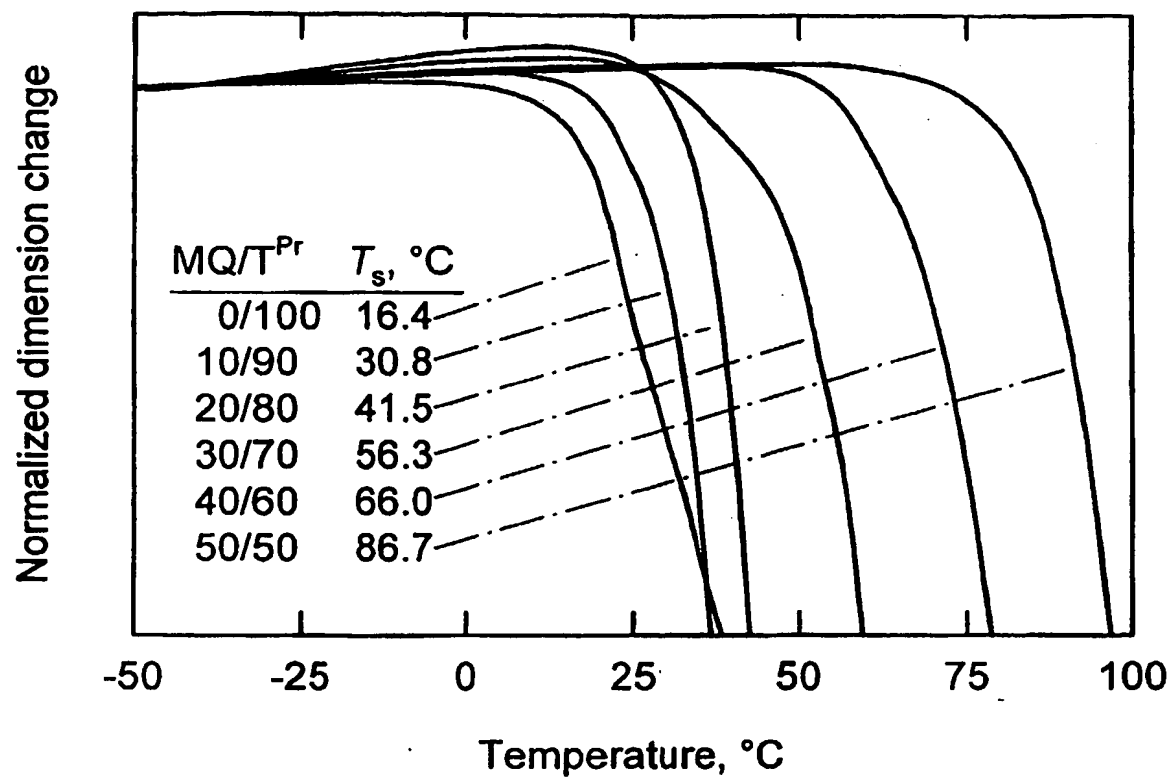
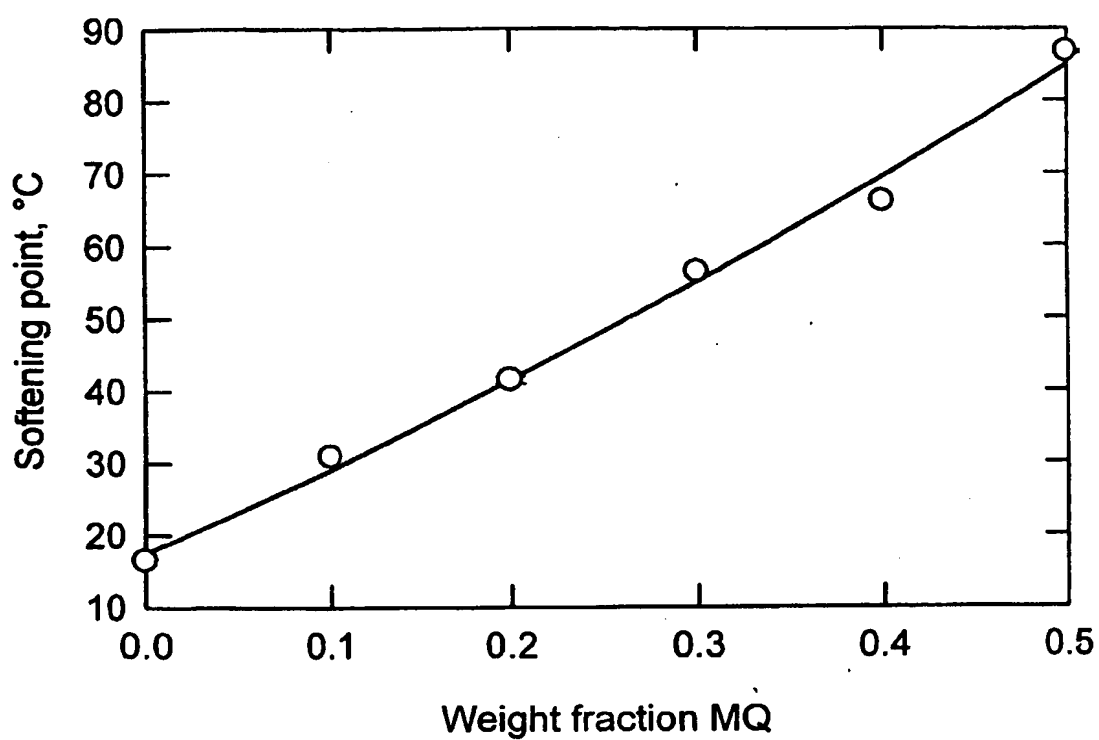


Figure 2



## INTERNATIONAL SEARCH REPORT

International Application No

PCT/US2005/003106

## A. CLASSIFICATION OF SUBJECT MATTER

IPC 7 C08L83/04 A61K7/11

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A61K C08L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, WPI Data, PAJ

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2002/031488 A1 (KANJI MOHAMED ET AL) 14 March 2002 (2002-03-14) paragraph '0016!; example 1	1-4
A	US 2003/236387 A1 (PAVLIN MARK S) 25 December 2003 (2003-12-25) paragraph '0214!	1-4

☐ Further documents are listed in the continuation of box C.☒ Patent family members are listed in annex.

## \* Special categories of cited documents :

- \*A\* document defining the general state of the art which is not considered to be of particular relevance
- \*E\* earlier document but published on or after the international filing date
- \*L\* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- \*O\* document referring to an oral disclosure, use, exhibition or other means
- \*P\* document published prior to the international filing date but later than the priority date claimed

- \*T\* later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- \*X\* document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- \*Y\* document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- \* & \* document member of the same patent family

Date of the actual completion of the international search

6 June 2005

Date of mailing of the international search report

17/06/2005

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2  
NL - 2280 HV Rijswijk  
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,  
Fax: (+31-70) 340-3016

Authorized officer

Buestrich, R

# INTERNATIONAL SEARCH REPORT

information on patent family members

International Application No

PCT/US2005/003106

Patent document cited in search report		Publication date	Patent family member(s)		Publication date
US 2002031488	A1	14-03-2002	JP	2002097366 A	02-04-2002
US 2003236387	A1	25-12-2003	US	2002187170 A1	12-12-2002
			CA	2447107 A1	21-11-2002
			EP	1392759 A1	03-03-2004
			JP	2004532324 T	21-10-2004
			MX	PA03010423 A	02-04-2004
			WO	02092663 A1	21-11-2002

## PCT

INTERNATIONAL PRELIMINARY REPORT ON PATENTABILITY  
(Chapter I of the Patent Cooperation Treaty)

(PCT Rule 44bis)

Applicant's or agent's file reference DC10008 PCT 1	<b>FOR FURTHER ACTION</b>		See item 4 below
International application No. PCT/US2005/003106	International filing date ( <i>day/month/year</i> ) 20 January 2005 (20.01.2005)	Priority date ( <i>day/month/year</i> ) 02 February 2004 (02.02.2004)	
International Patent Classification (8th edition unless older edition indicated) See relevant information in Form PCT/ISA/237			
Applicant DOW CORNING CORPORATION			

1. This international preliminary report on patentability (Chapter I) is issued by the International Bureau on behalf of the International Searching Authority under Rule 44 bis.1(a).
2. This REPORT consists of a total of 6 sheets, including this cover sheet.  
  
In the attached sheets, any reference to the written opinion of the International Searching Authority should be read as a reference to the international preliminary report on patentability (Chapter I) instead.

3. This report contains indications relating to the following items:

- |                                     |              |   |
|-------------------------------------|--------------|---|
| <input checked="" type="checkbox"/> | Box No. I    | Basis of the report   |
| <input type="checkbox"/>            | Box No. II   | Priority  |
| <input type="checkbox"/>            | Box No. III  | Non-establishment of opinion with regard to novelty, inventive step and industrial applicability  |
| <input type="checkbox"/>            | Box No. IV   | Lack of unity of invention  |
| <input checked="" type="checkbox"/> | Box No. V    | Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement |
| <input type="checkbox"/>            | Box No. VI   | Certain documents cited   |
| <input type="checkbox"/>            | Box No. VII  | Certain defects in the international application  |
| <input type="checkbox"/>            | Box No. VIII | Certain observations on the international application   |

4. The International Bureau will communicate this report to designated Offices in accordance with Rules 44bis.3(c) and 93bis.1 but not, except where the applicant makes an express request under Article 23(2), before the expiration of 30 months from the priority date (Rule 44bis .2).

The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland  Facsimile No. +41 22 338 82 70	Date of issuance of this report 07 August 2006 (07.08.2006)
	Authorized officer  Beate Giffo-Schmitt  e-mail: pt03@wipo.int



2

## PATENT COOPERATION TREATY

REC'D 15 JUN 2005

From the  
INTERNATIONAL SEARCHING AUTHORITY

WIPO PCT

PCT

To:

see form PCT/ISA/220

18/8

WRITTEN OPINION OF THE  
INTERNATIONAL SEARCHING AUTHORITY  
(PCT Rule 43bis.1)

Date of mailing

(day/month/year) see form PCT/ISA/210 (second sheet)

Applicant's or agent's file reference  
see form PCT/ISA/220**FOR FURTHER ACTION**  
See paragraph 2 belowInternational application No.  
PCT/US2005/003106International filing date (day/month/year)  
20.01.2005Priority date (day/month/year)  
02.02.2004International Patent Classification (IPC) or both national classification and IPC  
C08L83/04, A61K7/11Applicant  
DOW CORNING CORPORATION

## 1. This opinion contains indications relating to the following items:

- ☒ Box No. I Basis of the opinion
- ☐ Box No. II Priority
- ☐ Box No. III Non-establishment of opinion with regard to novelty, inventive step and Industrial applicability
- ☐ Box No. IV Lack of unity of invention
- ☒ Box No. V Reasoned statement under Rule 43bis.1(a)(i) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- ☐ Box No. VI Certain documents cited
- ☐ Box No. VII Certain defects in the international application
- ☐ Box No. VIII Certain observations on the international application

## 2. FURTHER ACTION

If a demand for international preliminary examination is made, this opinion will usually be considered to be a written opinion of the International Preliminary Examining Authority ("IPEA"). However, this does not apply where the applicant chooses an Authority other than this one to be the IPEA and the chosen IPEA has notified the International Bureau under Rule 66.1b/s(b) that written opinions of this International Searching Authority will not be so considered.

If this opinion is, as provided above, considered to be a written opinion of the IPEA, the applicant is invited to submit to the IPEA a written reply together, where appropriate, with amendments, before the expiration of three months from the date of mailing of Form PCT/ISA/220 or before the expiration of 22 months from the priority date, whichever expires later.

For further options, see Form PCT/ISA/220.

## 3. For further details, see notes to Form PCT/ISA/220.

Name and mailing address of the ISA:



European Patent Office  
D-80298 Munich  
Tel. +49 89 2399 - 0 Tx: 523656 epmu d  
Fax: +49 89 2399 - 4465

Authorized Officer

Buestrich, R

Telephone No. +49 89 2399-7473



**WRITTEN OPINION OF THE  
INTERNATIONAL SEARCHING AUTHORITY**

International application No.  
PCT/US2005/003106

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**Box No. I Basis of the opinion**

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1. With regard to the **language**, this opinion has been established on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.
  - ☐ This opinion has been established on the basis of a translation from the original language into the following language , which is the language of a translation furnished for the purposes of international search (under Rules 12.3 and 23.1(b)).
2. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application and necessary to the claimed invention, this opinion has been established on the basis of:
  - a. type of material:
    - ☐ a sequence listing
    - ☐ table(s) related to the sequence listing
  - b. format of material:
    - ☐ in written format
    - ☐ in computer readable form
  - c. time of filing/furnishing:
    - ☐ contained in the international application as filed.
    - ☐ filed together with the international application in computer readable form.
    - ☐ furnished subsequently to this Authority for the purposes of search.
3. ☐ In addition, in the case that more than one version or copy of a sequence listing and/or table relating thereto has been filed or furnished, the required statements that the information in the subsequent or additional copies is identical to that in the application as filed or does not go beyond the application as filed, as appropriate, were furnished.
4. Additional comments:

**WRITTEN OPINION OF THE  
INTERNATIONAL SEARCHING AUTHORITY**

International application No.  
PCT/US2005/003106

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**Box No. V Reasoned statement under Rule 43b/s.1(a)(i) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

---

**1. Statement**

Novelty (N)	Yes: Claims	1-4
	No: Claims	
Inventive step (IS)	Yes: Claims	1-4
	No: Claims	
Industrial applicability (IA)	Yes: Claims	1-4
	No: Claims	

**2. Citations and explanations**

**see separate sheet**

**Re item V:**

- 1 The following documents are referred to in this communication:

D1: US 2002/031488 A1 (KANJI MOHAMED ET AL) 14 March 2002 (2002-03-14)  
D2: US 2003/236387 A1 (PAVLIN MARK S) 25 December 2003 (2003-12-25)

- 2 The present application meets the criteria of Article 33(1) PCT, because the subject-matter of independent claim 1 and 2 is new in the sense of Article 33(2) PCT. Document D1 discloses a personal care product comprising a siloxane resin composition obtained by mixing:

- A) a MQ resin and
- B) a methyl silsesquioxane resin (cf. D1, paragraph 0016 and example 1).

The subject-matter of claim 1 and 2 of the present application differs from D1 in that for component B) a propylsilsesquioxane resin is disclosed. Therefore the subject-matter of claim 1 and 2 is novel over D1.

- 3 The present application does not meet the criteria of Article 33(1) PCT, because the subject-matter of independent claims 1 and 2 is not inventive in the sense of Article 33(3) PCT.

Document D1 discloses similar compositions for personal care applications and is therefor regarded as closest prior art.

The subject-matter of claim 1 and 2 of the present application differs from D1 in that for component B) a propylsilsesquioxane resin is disclosed.

The objective technical problem of the present application is to provide siloxane resins which show comparable extended wear and transfer resistance and improved gloss and which are non-tacky. Furthermore, there is a need for resins in hair care formulations that improve the curl retention properties of hair following treatment. It is not shown in the examples that a mixture of MQ and T propyl resins show improved properties with respect to the mixture of MQ and T methyl resins disclosed in D1.

Therefore the subject-matter of claims 1 and 2 is not inventive over D1.

**WRITTEN OPINION OF THE  
INTERNATIONAL SEARCHING  
AUTHORITY (SEPARATE SHEET)**

International application No.

PCT/US2005/003106

- 4 Dependent claims 3 and 4 do not contain any features which, in combination with the features of any claim to which they refer, meet the requirements of the PCT in respect of novelty and/or inventive step.

**Box No. VIII (iv) DECLARATION: INVENTORSHIP** (only for the purposes of the designation of the United States of America)

The declaration must conform to the following standardized wording provided for in Section 214; see Notes to Boxes Nos. VIII, VIII (i) to (v) (in general) and the specific Notes to Box No. VIII (iv). If this Box is not used, this sheet should

**Declaration of inventorship (Rules 4.17(iv) and 51b/s.1(a)(iv))  
for the purposes of the designation of the United States of America:**

I hereby declare that I believe I am the original, first and sole (if only one inventor is listed below) or joint (if more than one inventor is listed below) inventor of the subject matter which is claimed and for which a patent is sought.

This declaration is directed to the international application of which it forms a part (if filing declaration with application).

This declaration is directed to international application ..... (if furnishing declaration pursuant to Rule 26ter).

I hereby declare that my residence, mailing address, and citizenship are as stated next to my name.

I hereby state that I have reviewed and understand the contents of the above-identified international application, including the claims of said application. I have identified in the request of said application, in compliance with PCT Rule 4.10, any claim to foreign priority, and I have identified below, under the heading "Prior Applications," by application number, country or Member of the World Trade Organization, day, month and year of filing, any application for a patent or inventor's certificate filed in a country other than the United States of America, including any PCT international application designating at least one country other than the United States of America, having a filing date before that of the application on which foreign priority is claimed.

Prior Applications: .....

I hereby acknowledge the duty to disclose information that is known by me to be material to patentability as defined by 37 C.F.R. §1.56, including for continuation-in-part applications, material information which became available between the filing date of the prior application and the PCT international filing date of the continuation-in-part application.


I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

**Name:** GORDON, Glenn Viaplana .....

**Residence:** Midland, Michigan 48642 US MI .....  
(city and either US state, if applicable, or country)

**Mailing Address:** 3714 Collingwood Street .....  
Midland, Michigan 48642 US .....

**Citizenship:** US .....

**Inventor's**  .....  
(if not contained in the request, or if declaration is corrected or added under Rule 26ter after the filing of the international application, The signature must be that of the inventor, not that of the agent)


**Date:** 19 JAN 2005 .....  
(of signature which is not contained in the request, or of the declaration that is corrected or added under Rule 26ter after the filing of the international application)

**Name:** SCHMIDT, Randall Gene .....

**Residence:** Midland, Michigan 48642 MI .....  
(city and either US state, if applicable, or country)

**Mailing Address:** 813 Honeysuckle Circle .....  
Midland, Michigan 48642 US .....

**Citizenship:** US .....

**Inventor's Signature:**  .....  
(if not contained in the request, or if declaration is corrected or added under Rule 26ter after the filing of the international application. The signature must be that of the inventor, not that of the agent)

**Date:** JAN 19 2005 .....  
(of signature which is not contained in the request, or of the declaration that is corrected or added under Rule 26ter after the filing of the international application)

☒ This declaration is continued on the following sheet, "Continuation of Box No. VIII (iv)".

**Continuation of Box No. VIII (i) to (v) DECLARATION**

*If the space is insufficient in any of Boxes Nos. VIII (i) to (v) to furnish all the information, including in the case where more than two inventors are to be named in Box No. VIII (iv), in such case, write "Continuation of Box No. VIII ..." (indicate the item number of the Box) and furnish the information in the same manner as required for the purposes of the Box in which the space was insufficient. If additional space is needed in respect of two or more declarations, a separate continuation box must be used for each such declaration. If this Box is not used, this sheet should not be included in the request.*

**CONTINUATION OF BOX NO. VIII (iv)**

3-00 Name: STARK-KASLEY, Lori Ann

Residence: Midland, Michigan 48642 US MI

Mailing Address: 2683 East Mier Road, Midland, Michigan 48642 US

Inventors Signature: *Lori Ann Stark-Kasley* Date: 1-19-05

4-00 Name: WIEBER, Gary Michael

Residence: Midland, Michigan 48642 US MI

Mailing Address: 4407 Huron Drive, Midland, Michigan 48642 US

Inventors Signature: *Gary Michael Wieber* Date: 1-19-05

**Box No. VIII (iii)      DECLARATION: ENTITLEMENT TO CLAIM PRIORITY**

*The declaration must conform to the standardized wording provided for in Section 213; see Notes to Boxes Nos. VIII, VIII (i) to (v) (in general) and the specific Notes to Box No. VIII (iii). If this Box is not used, this sheet should not be included in the request.*

Declaration as to the applicant's entitlement, as at the international filing date, to claim the priority of the earlier application specified below, where the applicant is not the applicant who filed the earlier application or where the applicant's name has changed since the filing of the earlier application (Rules 4.17(iii) and 51bis.1(a)(iii)):

I relation to this International application,

DOW CORNING CORPORATION, is entitled to claim priority of earlier application

Number US 60/541,001

by virtue of the following:

DOW CORNING CORPORATION is entitled as employer of the inventor

GORDON, Glenn Viaplana  
SCHMIDT, Randall Gene  
STARK-KASLEY, Lori Ann  
WIEBER, Gary Michael

This declaration is made for the purposes for all designations, except the designation of the United States of America.

☐ This declaration is continued on the following sheet, "Continuation of Box No. VIII (iii)".



**Box No. VIII (ii)      DECLARATION: ENTITLEMENT TO APPLY FOR AND BE GRANTED A PATENT**

*The declaration must conform to the standardized wording provided for in Section 212; see Notes to Boxes Nos. VIII, VIII (i) to (v) (in general) and the specific Notes to Box No. VIII (ii). If this Box is not used, this sheet should not be included in the request.*

Declaration as to the applicant's entitlement, as at the international filing date, to apply for and be granted a patent (Rules 4.17(ii) and 51 bis.1(a)(ii)), in a case where the declaration under Rule 4.17(iv) is not appropriate:

In relation to this International application,

DOW CORNING CORPORATION is entitled to apply for and be granted a patent by virtue of the following:

DOW CORNING CORPORATION is entitled as employer of the inventor,

GORDON, Glenn Viaplana of 3714 Collingwood Street, Midland, Michigan 48640, US  
SCHMIDT, Randall Gene of 813 Honeysuckle Circle, Midland, Michigan 48642, US  
STARK-KASLEY, Lori Ann of 2683 East Mier Road, Midland, Michigan 48642, US  
WIEBER, Gary Michael of 4407 Huron Drive, Midland, Michigan 48642, US

This declaration is made for the purposes for all designations.

☐ This declaration is continued on the following sheet, "Continuation of Box No. VIII (ii)".